### INDUSTRIAL TECHNOLOGIES PROGRAM

### **Parallel Beam X-Ray Diffraction System**

# **Advanced X-Ray Optics Enable On-Line Steel Phase Measurement**

X-Ray Optical Systems is offering the first on-line x-ray diffraction (XRD) system suitable for measuring steel composition and phase in industrial environments. Created with funding assistance from the U.S. Department of Energy, the new XRD system has been successfully tested in a steel galvannealing line to monitor crystalline properties in real time during production. The system uses advanced x-ray optics to perform measurements typically done off line in the lab, allowing manufacturers to maintain product quality and consistency more effectively.

Real-time, nondestructive measurement of material properties on line represents the state of the art in industrial control. With the incorporation of x-ray optics, x-ray diffraction technology can identify structural phases, determine grain size, and measure stress and crystal orientation ("texture") of materials on line. Although XRD technology

is widely used in the lab to determine these properties, existing systems have proven too bulky and complex for on-line monitoring.

X-Ray Optical Systems' new X-Beam® technology is a compact, portable, low-power system that can be used to determine and monitor surface-phase composition on line in harsh industrial settings. In addition to offering real-time capability, the technology eliminates misalignment and common instrument error functions. The powerful x-ray optics technology used in this system may also enable the development of other diffraction-based sensors for industry because of its low power requirements, lack of moving parts, and insensitivity to sample position, temperature changes, and vibration.

In 2004, the developer received an "R&D 100" award for a new application of the DOE-sponsored technology, measuring the crystal orientation of superconducting cable.



## **Benefits for Our Industries and Our Nation**

The XRD technology provides the first online diffraction-based tool for measuring structural phases, grain size, stress, and materials texture. The technology's benefits relative to current XRD technology include portability, increased x-ray intensity (up to 100 times), decreased power consumption, improved measurement efficiency, and reduced system sensitivity to sample displacement and general defocusing errors.

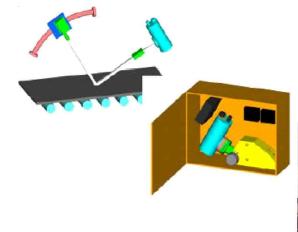
### **Applications in Our Nation's Industries**

The initial focus of the X-Beam® technology is characterization and control of the galvannealing process. Other applications in the steel industry include:

- · measurement of retained austenite
- mineral analysis for incoming raw materials
- · stress analysis in electrical sheets
- · measurement of grain size
- measurement of surface coatings, contamination, oxides, and corrosion products

#### Other applications for on-line xray diffraction measurement may include:

- aluminum (texture and alloy phase)
- paper products (fiber orientation)
- cement (component phase, free lime content, particle size)
- semiconductor (wafer orientation, stress, and texture)
- pharmaceuticals (component quantity and structure)
- machined parts (residual stress)
- ceramics (grain size)





Evolution from original solid model to (nearly) final product.

#### **Project Summary**

#### **Objective:**

The project objective was to develop a compact, on-line x-ray diffraction system to measure material composition and demonstrate it in an industrial environment. Specifically, the researchers designed, built, and tested an on-line XRD sensor system for phase measurement and composition control on a steel galvannealing line at AK Steel. This application was chosen because the quality of galvanneal coatings must be consistently high in order for these steels to withstand subsequent bending, stamping, shaping, and painting.

#### Technology:

Most conventional x-ray powder diffractometers use a parafocusing Bragg-Brentano geometry, which allows for analysis with high resolution and high beam intensity but requires well-aligned and carefully prepared samples. To obtain accurate data, for example, the source-to-sample distance must be constant and equal to the sample to-detector distance at all times. These constraints make conventional technology impractical for on-line use.

The X-Beam® system is composed of an appropriately matched capillary-optic and x-ray source combination, detector, and post-sample anti-scatter optic system. The technology uses capillary x-ray optics to collect x-rays over a large solid angle from a low-power source and form an intense parallel beam. With parallel geometry and high beam intensity, sample position can vary and source-to-sample distance does not have to equal sample-to-detector distance. This geometric flexibility can accommodate existing manufacturing conditions and a much broader range of sample shapes and sizes.

Other features of the X-Beam® include:

- Pre-aligned source and optics
- Plug-and-play operation
- Simple integration with the sensor package
- Consistent performance over an extended temperature range/integrated thermal stability control
- · Integrated microprocessor

#### **Project Milestones:**

This project was awarded in July 1999 through a Small Business Innovation Research (SBIR) solicitation by the Sensors and Controls Program. Key tasks and milestones met were:

- Concept feasibility proven July 2000
- X-ray system first assembled August 2002
- On-line installation simulated September 2002
- X-ray system tested on a commercial galvanneal line October 2002

#### **Commercialization:**

The system is available commercially from X-Ray Optical Systems, Inc., a leading manufacturer of x-ray optics, components and analyzers serving OEMs and end- users in the analytical instruments, petroleum, pharmaceutical, and semiconductor industries. Please visit the X-Ray Optical Systems website at <a href="http://www.xos.com/xbeam.htm">http://www.xos.com/xbeam.htm</a> for more information regarding the X-Beam® product line.

#### **For More Information**

Please contact Tom Bievenue of X-Ray Optical Systems, Inc., at 518-464-3334 (ext. 702) or <a href="mailto:tbievenue@xos.com">tbievenue@xos.com</a>.

#### **Project Partners**

X-Ray Optical Systems, Inc. East Greenbush, NY

AK Steel Ashland, KY

John Stubbles Mason, OH

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